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DEVELOPMENT DEPARTMENT

QUARTERLY PROGRESS REPORT #6

October 31, 1962 - January 31, 1963

Synthesis of Regulated Structure
Polyphenylether-Siloxane Block Copolymers

Contract # DA-19-020-ORD-5507



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A B S T R A C T

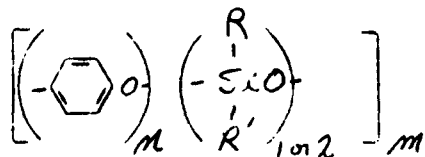
The large scale high temperature alkaline hydrolysis reaction was worked up and of several specialized techniques employed to purify the final product, one was very successful. The catalyst concentration and form was found to be a major factor in this reaction and a factorial experiment was begun to study this and four other parameters.

Tetrahydrofuran was found to be a successful solvent for the measurement of molecular weights of monomers and polymers using the Vapor Pressure Osmometer. Thin layer chromatography has been instituted as a successful analytical tool to expedite this program.

A literature search uncovered no new specific reference to the synthesis of 4,4'-dihydroxypolyphenylethers or p,p'-dihydroxypolyphenylethers, but did focus attention on a phenol via cumene route which holds promise as an adaptable technique.

I. Introduction

This contract is directed toward the synthesis of high temperature elastomers consisting of regulated structure, polyphenylether siloxane block copolymers of the type:



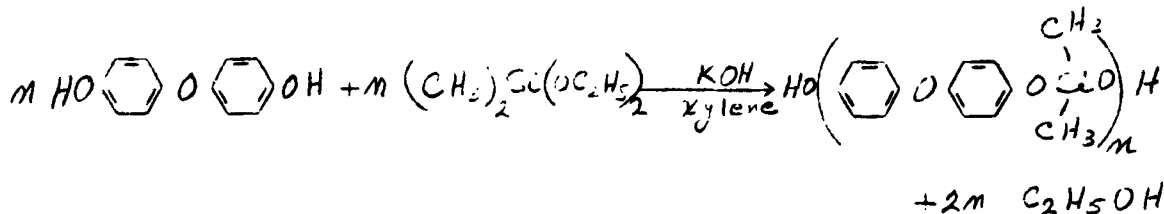
where:

$$n = 2, 3, \dots 10\dots$$

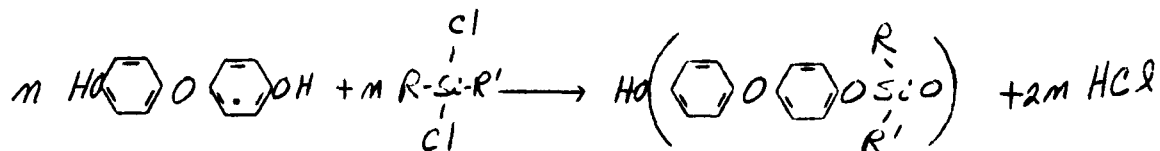
$$m = 100, \dots 300\dots$$

R & R' are alkyl, aryl, or vinyl groups

The principle effort thus far has been directed toward the synthesis of the monomer units. This having been done, a number of polymerizations have been carried out. Two polymerizations were carried out by an alcoholysis condensation reaction of the type:



The other polymerizations were carried out as direct condensations between the chlorosilanes and dihydroxydiphenylethers, for example:



It is confirmed that these polymers thus far produced are of too low a molecular weight to be of any value as elastomers. Because these polymerizations are condensation reactions capable of undergoing rearrangement, the length of the polymer chain is sensitive to the stoichiometry of the reaction mixture, and, more important, to the presence of any "chain stopping" derivatives of the monomers. Logically, then, the immediate aim of the program has been to obtain pure monomers.

Painstaking effort has demonstrated the nature of the impurities that can be present in the dihydroxydiphenylether and how to separate them. In addition, the problem is being approached at the source: the alkaline hydrolysis reaction for the synthesis of this monomer. A factorial experiment has been initiated to determine those reaction conditions that will produce the cleanest yield. The technique of thin layer chromatography has been introduced as a means of ascertaining purity of the product and as a means of expediting the assay of the reaction series.

For the same purpose, a literature search was conducted to seek out any new synthetic techniques for the p,p'-dihydroxypolyphenylether series that may have been published since the search conducted at the start of the program. There was only partial success here in that the copious references to a phenol via cumene process may be adaptable to the synthesis of these monomers.

II. Synthesis & Purification of 4,4'-Dihydroxydiphenylether (DHDPE)

As mentioned in Quarterly Progress Report #5, a 20-gallon high temperature alkaline hydrolysis reaction was run. This reaction, designated # HPL-20 was run first without catalyst, resulting in no apparent yield as determined by an assay of a reaction mixture aliquot. An aqueous solution of the cupric sulfate catalyst was prepared containing 0.1 kg. of Cu^{++} in the 1/2-gallon of solution. This catalyst solution was added to the reaction mixture with stirring and the reaction was rerun using the same conditions of 185°C. for 6 hours.

A. Workup of Large Scale Reaction # HPL-20

1. Workup of Crude Reaction Mixture

FIGURE I illustrates the workup of the large scale reaction and of the major fractions obtained. The dark reaction mixture was filtered leaving only a small residue (a) instead of an expected very large residue. This creates a sharp inconsistency with a previous reaction, Y51*, run under the same conditions in a one liter autoclave and yielding a corresponding residue fraction essentially composed of 36% of the 4,4'-dibromodiphenylether starting material. Again, in contrast, a yellow oil fraction (b) was separated at this point in the workup of the large scale reaction, identified by its infrared spectrum as being largely p-bromodiphenylether. A Zeisel ether cleavage

* Erroneously reported as Y49 in Quarterly Progress Report #5, page 7.

test on this oil confirms also the presence of isopropoxy substituted groups. In addition, thin layer chromatography indicates that there are at least two compounds present in this oil.

In continuing the workup above, the filtrate was neutralized with concentrated hydrochloric acid and then extracted with ether. The brown ether layer was boiled with toluene to remove the ether in order to recrystallize the desired product from toluene. Part of the ether soluble material was insoluble in the toluene, (c) but subsequent extraction (soxhlet) with benzene followed by recrystallization yielded red crystalline aggregates identified by infrared spectra as being DHDPE. The colored impurities in this fraction were almost completely removed by extracting an alkaline aqueous solution of this material with chloroform, after which the desired product was extracted from the neutralized aqueous solution with ether. Further colored impurities were removed by passing the ether solution through activated alumina and by treating the solution with activated charcoal. Recrystallization from toluene yielded a white crystalline product. It should be noted here that the alumina had evidently held back a considerable portion of the fraction. Elution of the alumina bed with acetone did not increase the yield.

2. Sublimation Purification

The purity of this desired product fraction, (D_1), was still questionable due to its broad melt point range, 163-169°C. In addition, its molecular weight measured in tetrahydrofuran was 197 instead of 202 ± 2 . A small scale experiment was set up to determine the feasibility of this method of purification for the 4,4'-dihydroxydiphenylether. A small amount of fraction (D_1) was placed at the bottom of a right angle shaped tube. The tube was evacuated and the end containing the 4,4'-dihydroxydiphenylether was placed in an oil bath maintained at 155°C. The experiment, carried on over a weekend resulted in a needle crystal deposit near the top of the tube and a liquid gray brown residue which solidified on cooling. The white needle crystals had a melt point of 130-160°C., a molecular weight of 211 in tetrahydrofuran. The infrared spectrum of these crystals indicated only 4,4'-dihydroxydiphenylether. A second trial with continuous vacuum supplied by the house vacuum resulted in a similar separation except that in the second trial the residue comprised a much greater fraction than in the first trial. In the second trial, the white crystalline portion had a melt point of 163-172°C., and a molecular weight of 111 measured in tetrahydrofuran. Its infrared spectrum indicated hydroquinone and some dihydroxydiphenylether.

In order to check the possibility that fraction D₁ contained hydroquinone, the material was washed vigorously with two separate liters of water. A hydroquinone determination run on the wash water gave a value of 0.02%, or 0.2 gm. total in the wash water. This figure is on the order of the solubility of 4,4'-dihydroxydiphenylether in water and it was therefore concluded that no hydroquinone was present in the D₁ fraction. The hydroxyl number (equivalent KOH) of fraction D₁ is 549.7 corresponding to a molecular weight of 204.

3. Purification of the Principle Fraction

Purification of the D₂, D₃ fractions by chloroform extraction of the alkaline aqueous solution was not successful as in the case of fraction D₁. The yellow ether extract of the neutralized aqueous solution could not be freed of contaminants by the activated alumina or charcoal. Recrystallizing the material from acetic acid resulted in a blue crystalline mass (D₄). The colored impurities were effectively removed by adding petroleum ether to a methyl ethyl ketone solution of the blue material until the cloud point was reached. The dark colored oily precipitate slowly settled out on the walls of the glass container leaving a light yellow colored solution. The deep black material recovered (0.5 gm.) could not be identified by infrared since its spectrum indicated essentially 4,4'-dihydroxydiphenylether.

The light tan crystals (D₅) resulting from the above purification were found to undergo a color change on oven drying at 110°C. The color change was back toward the blue which was believed to have been removed.

4. Zone Refining

Two attempts to purify 4,4'-dihydroxydiphenylether using a Zone Refining apparatus were unsuccessful. The first trial was performed on a mixture of materials from previous experiments. The second trial was performed on a sample of the partially reverted (oven dried) material from fraction (D₅) in a sealed tube. The experiment was carried on for three days or a total of about twelve passes with a melt zone speed of two inches per hour. The glass tube was mounted in a horizontal position. On melting, the material turned dark brown. During the experiment white needle crystals condensed on the uppermost walls of the tube with the heaviest concentration toward the "impurity" end of the tube (end at which melt zone completed travel). Samples of the brown fused material taken from both ends of the tube had the following melt points:

1. "Impurity" End - M.P. 112-128°C.
2. Refined End - M.P. 125-138°C.
3. White needle crystals - M.P. 162-174°C., soluble in water which confirms it as hydroquinone

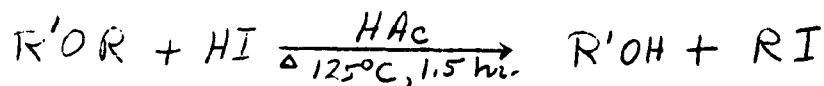
The starting material had a melt point range of 162-170°C.

5. Final Purification

Fraction (D₅) was successfully purified by hot filtration of its water solution which separated off an immiscible brown oil. Since activated charcoal was used to decolorize the aqueous solution, no attempt was made to collect the minute amounts of the brown oil. The desired pure white product recrystallized from solution on cooling after which it was extracted from the aqueous medium with ether. The product was precipitated from the ether solution with petroleum ether. It was observed here that the product was still susceptible to color change during oven drying unless the final ether solution was dried over Drierite before precipitating the product. This then was the procedure followed. After oven drying at 105°C. for 24 hours, there was no evidence of color change in the product. However, after letting the product stand for about two weeks (covered jars), a slight color change (tan) did take place when the product was redried for 48 hours at 110°C. The indication here is that the product should be reprecipitated from "dry" ether and then stored in a desiccator.

6. Alkoxy Substituted Diphenylethers


The two liquid oily fractions, (B) and (E), were shown to contain alkoxy (isopropoxy) substituted groups by a qualitative Zeisel Ether Cleavage test. Since these are sizeable fractions, it was important to determine their potential in DHDPE that might be obtained by converting the alkoxy groups to hydroxyl groups. Small scale reactions were run patterned after the Zeisel method:



where:

R' = aryl group

R = alkyl group

Workup of the reaction using fraction (B) yielded no DHDPE. Evidently fraction (B) has much mono-substitution of the type: . This probability is also consistent with the infrared spectrum of fraction (B).

Workup of the reaction using fraction (E) yielded an amount of 4,4'-dihydroxydiphenylether equivalent to three percent of the total theoretical yield of the 20-gallon reaction, 100 gm.

B. Study of Reaction Parameters in High Temperature Alkaline Hydrolysis Reaction

1. Anomalous Catalyst Behavior

It has been observed in the past that high temperature alkaline hydrolysis reactions run under apparently identical conditions produced inconsistent results. A striking example of this is reaction Y54 and reaction HPL-20 run initially without catalyst. Another example is reaction Y51 and reaction HPL-20 run with catalyst present. Reaction Y49 and Y51 (without catalyst) were run in the one liter stainless steel autoclave during a series of similar reactions. Reactions HPL-20 (first without and then with catalyst) were run in a 20-gallon stainless steel autoclave.

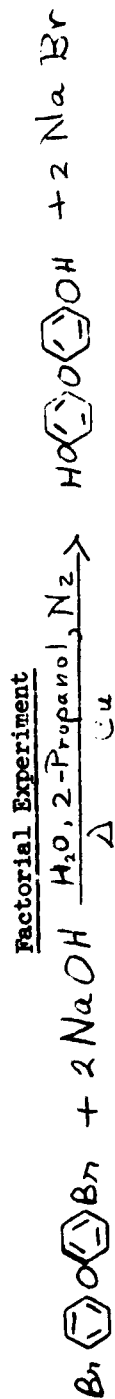
<u>Reactions</u>	<u>Conditions</u>	<u>Catalyst</u>	<u>Yield Desired Product</u>	<u>Starting Material Reclaimed</u>	<u>Other Products</u>
HPL-20 (Initial)	Same	No	0%	100%	
Y54		No	50%	20%	
HPL-20 (Final)	Same	Yes	30%	3%	Immiscible Oil
Y51		Yes	41%	37%	No Immiscible Oil

It is evident here that not only is the catalyst concentration a major factor in the reaction, but its form during the reaction. Also, "contamination" or "conditioning" of the reaction vessel by the catalyst is also in evidence according to the data. Only on this basis can the high yield in reaction Y54 be explained since it was run in a vessel "preconditioned" during previous reactions.

2. Factorial Experiment

A factorial experiment was designed for the high temperature alkaline hydrolysis reaction to study the parameters of time, temperature, catalyst concentration, starting material concentration, and alcohol (2-propanol) concentration. Eight of the possible thirty-two permutations were chosen for an initial study. The first two were run in error, but are included in TABLE I as 1 and 2 although they were rerun correctly as 3 and 4 respectively. In addition, a pretrial run was made (designated 0) without catalyst as a check on experiment Y54 assuming that any residual catalyst would have been removed during the use made of the one liter autoclave for other unrelated experiments just prior to this present series. Also, a final run without catalyst (designated 11) was scheduled to test for any preconditioning effects due to the catalyst during the series of runs. The conditions for this sequence of experiments are given in TABLE I.

TABLE I



Factorial Experiment

Exp. Sequence	Exp. #	Factorial Series #	m NaOH m DBDPE	Temp. °C.	cc. Alcohol cc. H ₂ O	m DBDPE l Sol.	Time Hrs.	m Catalyst (in Cu ++) m DBDPE
0	Y59		4.38	185	1	1/3	6	0
1	Y60		8.76	210	1/3	1/6	3	.0188
2	Y61		8.76	210	1	1/3	6	.0188
3	Y62	2	4.38	210	1/3	1/3	3	.0188
4	Y63	16	4.38	210	1	2/3	6	.0188
5	Y64	21	4.38	185	1/3	2/3	3	.0944
6	Y65	27	4.38	185	1	1/3	6	.0944
7	Y66	3	4.38	185	1	1/3	3	.0188
8	Y67	13	4.38	185	1/3	2/3	6	.0188
9	Y68	24	4.38	210	1	2/3	3	.0944
10	Y69	10	4.38	210	1/3	1/3	6	.0188
11	Y70		4.38	185	1	1/3	6	0

The reaction mixtures were prepared by dissolving the cupric sulfate catalyst in the sodium hydroxide solution. To this was added the 2-propanol. The 4,4'-dibromodiphenylether was added to the solution when the solution was introduced into the autoclave. The resulting solutions differed markedly from one another according to the concentrations of NaOH, catalyst, and alcohol. Observations made on these solutions are listed in TABLE II.

TABLE II
Pre-reaction Mixtures

Exp. #	<u>gm NaOH</u> <u>l. Sol.</u>	<u>cc. Alcohol</u> <u>cc. H₂O</u>	<u>gm CuSO₄.5 H₂O</u> <u>l. Sol.</u>	Observations
Y60	58.4	1/3	0.785	Homogeneous light blue solution.
Y61	58.4	1	1.571	Separate colorless alcohol & homogeneous blue aqueous phases.
Y62	58.4	1/3	1.571	Homogeneous blue solution-dark blue gelatinous ppt. formed on standing.
Y63	116.8	1	3.142	Similar to #2.
Y64	116.8	1/3	15.71	Separate colorless alcohol phase with blue black ppt. in aqueous phase → black ppt.
Y65	58.4	1	7.858	Separate colorless alcohol phase- on standing blue black gelatinous ppt. forms in aqueous phase.
Y66	58.4	1	1.571	Similar to #2.
Y67	116.8	1/3	3.142	Similar to #5-ppt. forms slower.
Y68	116.8	1	15.71	Separate colorless alcohol phase-ppt. forms as large black aggregates.
Y69	58.4	1/3	1.571	Homogeneous blue solution.

The autoclave vessel was inspected after each of the first four runs. After run Y60 it was noted that loose reduced copper granules had deposited on the welded joints of the stainless steel stirrer and were removed easily by brushing. After run Y61, in addition to the granules, copper had begun to plate out on the vessel walls. The only treatment found at all successful for removing the copper plating was dilute HCl solution. Dilute HCl solution is not compatible with stainless steel and it was decided to forego this treatment which would probably require several hours of exposure each time to remove the plating. The treatment therefore was limited to a good scrubbing of the autoclave to remove the loose granules. It was also expected that run Y70 (no catalyst) would afford a measure of the activity of the plated copper on the reaction.

During the series of runs a unique characteristic was observed in those reactions in which a greater concentration of NaOH was used. In reactions Y63, Y64, Y67, and Y68, after cooling down, the vessel pressure was about a hundred psi greater than the starting pressure indicating that about 0.1 moles of a gaseous product had formed during the reaction.

The reactions are to be assayed using the techniques of thin layer chromatography which are described later in this report.

C. Analytical Methods & Properties of 4,4'-Dihydroxydiphenylether

1. Calibration of Vapor Phase Osmometer & Measurements

The Mechrolab Model 301A Vapor Phase Osmometer was obtained and calibrated using benzene and tetrahydrofuran. The VPO was calibrated for benzene with benzil and biphenyl. However, the polyphenylether siloxane copolymers were found to have too low a solubility for measurements to be made in benzene.

The VPO was therefore calibrated for tetrahydrofuran with diphenylether, p,p'-biphenol, and 4,4'-dibromodiphenylether as solutes. The calibration with tetrahydrofuran was expected to be difficult due to the volatility of the solvent and it was. However, after three calibration trials, the technique was perfected so that reproducibility among the three materials used was better than 1%. In addition, it was found that with tetrahydrofuran, the reproducibility was independent of the time intervals (1, 2, or 3 min.) at which readings were taken after depositing the sample solution. It can be said, therefore, that equilibrium was established almost immediately.

In each case, R/C was plotted for four solution concentrations ranging from 0.02 m/l to 0.1 m/l, where R is the bridge balance reading in ohms and C is the solution concentration.

In measuring monomers, where the slope of R/C is expected to be constant, as it was in the case of the three calibration solutes, the molecular weight of the unknown could be found by:

$$\text{Mol. Wt.} = C_g/C_m$$

Where: C_g = concentration in gms/liter and
 C_m = concentration in moles/liter

as determined from the calibration plot according to the measured value R .

In measuring polymers, for which R/C is not constant, it is necessary to plot R/C versus C and extrapolate to find the intercept at $C = 0$. In determinations on polymers, therefore, it is necessary to take readings of a series of concentrations.

The molecular weight could then be found by:

$$\text{Mol. Wt.} = K_c/K_x$$

where: K_c = calibration constant = $\lim_{C_m \rightarrow 0} \left(\frac{R}{C_m} \right)$ and
 for the known calibration solute

$$K_x = \lim_{C_g \rightarrow 0} \left(\frac{R}{C_g} \right)$$

for the unknown solute.

In the case of tetrahydrofuran, the calibration slopes of R/C versus C were zero within the tolerance of the instrument. This indicates that tetrahydrofuran is an ideal solvent for these types of materials and should give excellent results as long as the carefully established technique is followed rigorously.

In the above manner, measurements were made on various materials originating from past alkaline hydrolysis reactions (some cited in this report) and on polymers reported in Quarterly Progress Report #5 - III, page 13.

The results of the polymer molecular weight determinations are given in TABLE III.

TABLE III

Molecular Weights of Polyphenylether Siloxane Copolymers

Polymer OB-734#	Phenyl- ether	Mole % Phenyl- ether	Mole % DPDCS	Mole % MVDCS	Molecular Wt.	Remarks
20A	DHDPE	51.3	43.7	5.0	1453	Min. Amt. Insoluble Material
20B	HQ	51.3	45.7	3.0	-	Polymer not Homogeneous - not Measured
20C	HQ	52.6	42.4	5.0	-	" " "
20D	DHDPE	52.6	44.4	3.0	404	Min. Amt. Insoluble Material
20E	HQ	51.3	43.7	5.0	323	" " " "
20F	DHDPE	51.3	45.7	3.0	1301	" " " "
20G	DHDPE	52.6	42.4	5.0	-	Polymer not Homogeneous - not Measured
20H	HQ	52.6	44.4	3.0	-	" " "
24A	DHDPE	47.6	47.6	4.8	-	No Reproducibility in Read- ings

A measurement made on Eastman's 4,4'-oxydiphenol gave a value of 197 which agrees precisely with a previous determination on this material by an independent laboratory.

2. Thin Layer Chromatography

Thin layer chromatography was instituted as an analytical and a preparative tool. It is ideally suited to the former, comparable to paper chromatography with the additional advantages of sharper separations, much shorter developing times, and enabling the use of corrosive indicator solutions. As a preparative tool, it is adequate for the isolation of micro-amounts of material for further analysis. In addition, the substrate material can also be used as a chromatography column packing to purify large amounts of material once the correct solvent has been determined by the thin layer techniques. With this method, it is expected that:

- a. The purity of the 4,4'-dihydroxydiphenylether monomer can be ascertained.
- b. The 4,4'-dihydroxydiphenylether monomer can be purified.
- c. Reactions can be assayed accurately and completely with the separation and identification of all the side products.

To date, several developing solutions of benzene and petroleum ether in varying proportions have been tried as well as benzene alone. These were tried on a number of materials originating from several past reactions. Of these developing solutions, benzene alone is the most successful. The results using benzene are shown in FIGURE II. The illustration represents the developed plate after the presence of material has been indicated by the destructive technique of charring with $\text{Cr}_2\text{O}_7^{2-}$ - H_2SO_4 solution. It will be noted by the Eastman 4,4'-oxydiphenol sample that benzene is not the proper solvent for separating 4,4'-dihydroxydiphenylether since the material was barely moved. It will be necessary to try other solvent systems.

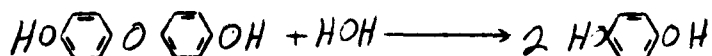
3. Sensitivity of 4,4'-Dihydroxydiphenylether to Moisture

Evidence has been accumulating pointing to the possibility that 4,4'-dihydroxydiphenylether may be sensitive to moisture, allowing under some conditions a slow oxidation of the product.

In the sublimation experiments reported in II A (2), the first experiment carried out in an evacuated (vacuum pump), sealed tube resulted in no or very little hydroquinone. The second experiment was carried out in a "closed system" where the vacuum was supplied continuously from house facilities. In this case, the vacuum is created by means of a steam jet which would allow a definite water vapor pressure in the sublimation vessel. Hydroquinone was one of the major products in this experiment.

The experience with the final purified product of reaction HPL-20 (II A(5)) showed that the product precipitated from water saturated ether was susceptible to discoloration on oven drying. However, the product, when precipitated from ether solution dried over Drierite, does not discolor on oven drying unless the product is allowed to stand for a few weeks. Then it again becomes susceptible to discoloration on oven drying.

It is not possible at present to determine whether this behavior is due to the catalytic action of an impurity but it is suggested here that the moisture is the agent causing the formation of hydroquinone which must form through the combination of water with 4,4'-dihydroxydiphenylether:



The discoloration is probably then due to the further oxidation of the hydroquinone.

4. Caustic Fusion of the p,p'-Disulfonylchloride of Diphenylether - Continued - Identification of Products

This reaction was reported in Quarterly Progress Report #5 (II B (3)). It was reported that 3 gms. of long needle crystals were isolated, having a melting point range of 237-243°C., and being soluble in alkaline aqueous solution. Additional data have since been gathered on this material.

Elemental Analysis

% C	-	58.83	58.63
% H	-	4.15	4.02
% S	-	12.90	12.90
% O	-	23.70	
% Cl	-	Trace	
Total		99.58	99.25

Disregarding the chlorine, the best approximation to the data is the elementary formula $C_{12}H_{10}SO_4$. This compound would have a molecular weight of 250 as compared to the measured molecular weight of 237. The infrared spectrum indicates an absence of both a phenolic OH and an ether linkage. The sulfonic acid group and both mono-substitution and 1,4-disubstitution are confirmed. This leads to the structure:



Molecular Weight - 234

Elementary Formula - $C_{12}H_{10}SO_3$

This structure is the one believed to be correct. Unfortunately, while the compound was cited in Beilstein, the melting point was not and this conclusion could not be corroborated or refuted. Also, the melting points of the other compounds possible according to the elemental analysis could not be found in the literature.

D. Literature Search

A search of the literature on the subject of the synthesis of 4,4'-dihydroxydiphenylether has not uncovered anything that was not already cited in the Quarterly Progress Report #1. However, during the search, it was observed that there are numerous references on the synthesis of phenol from cumene via the hydroperoxide intermediate. In one case, resorcinol was synthesized from m-diisopropylbenzene. It is apparent that this synthetic technique has gained much momentum in development and as an industrial process. The process at this point appears to have excellent potential as a route to dihydroxydiphenylethers and dihydroxypolyphenylethers. If any drawback can be contemplated, it is probably the expected side products that can occur and which would have to be separated. This consideration places the cumene process about on par with the present alkaline hydrolysis synthesis and unless no progress is forthcoming in the latter, the former should be considered.

III. Conclusions

Neither sublimation nor zone refining are feasible techniques for the purification of 4,4'-dihydroxydiphenylether. The technique of precipitating the product from "dry" ether after recrystallization from hot water is to date the only successful technique. The pure product will then be heat stable as long as it is kept moisture free.

The catalyst concentration and form is a major factor in the high temperature alkaline hydrolysis reaction. Moreover, the catalyst causes a certain amount of conditioning or contamination of the stainless steel reaction vessel. This effect may be responsible for the poor reproducibility of past reactions.

IV. Projected Work

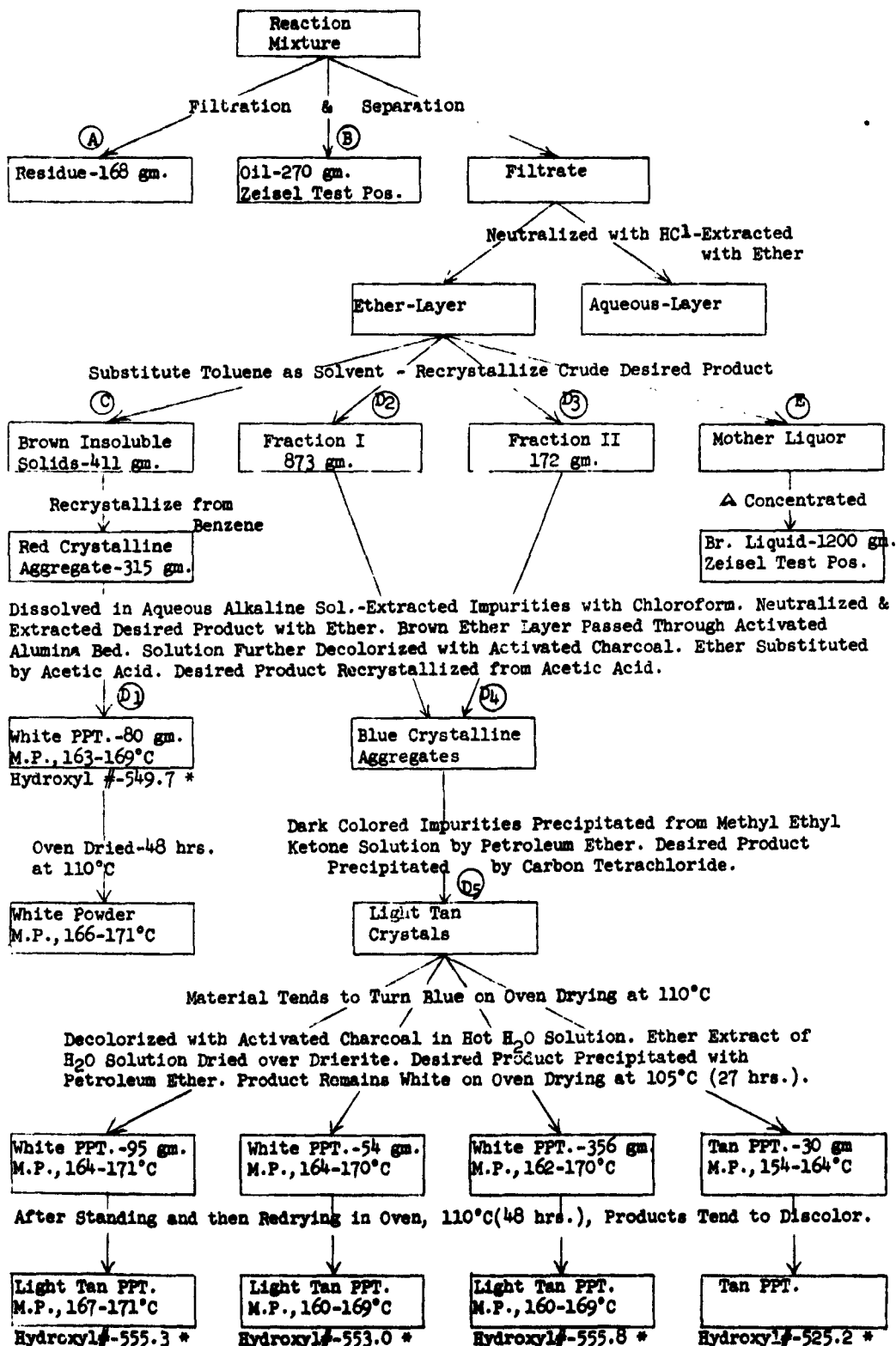
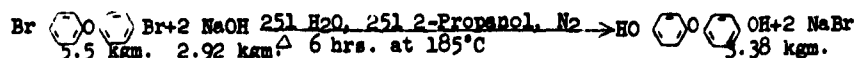
1. Repurify, if necessary, the pure product of the 20-gallon reaction.
2. Purify, by distillation, the diphenyldichlorosilane and the methylvinylchlorosilane monomers.
3. Continue polymerization reactions using the pure monomer materials.
4. Assay the factorial reaction series using thin layer chromatographic techniques.
5. Run physical measurements on new polymers synthesized.
6. Crosslink new polymers synthesized.

Manhours Expended, January 1963

R. MacFarlane, Jr.	56 hours
E. S. Yankura	160 hours

FIGURE I

Workup of Large Scale Alkaline Hydrolysis-EPL-20(Y56)



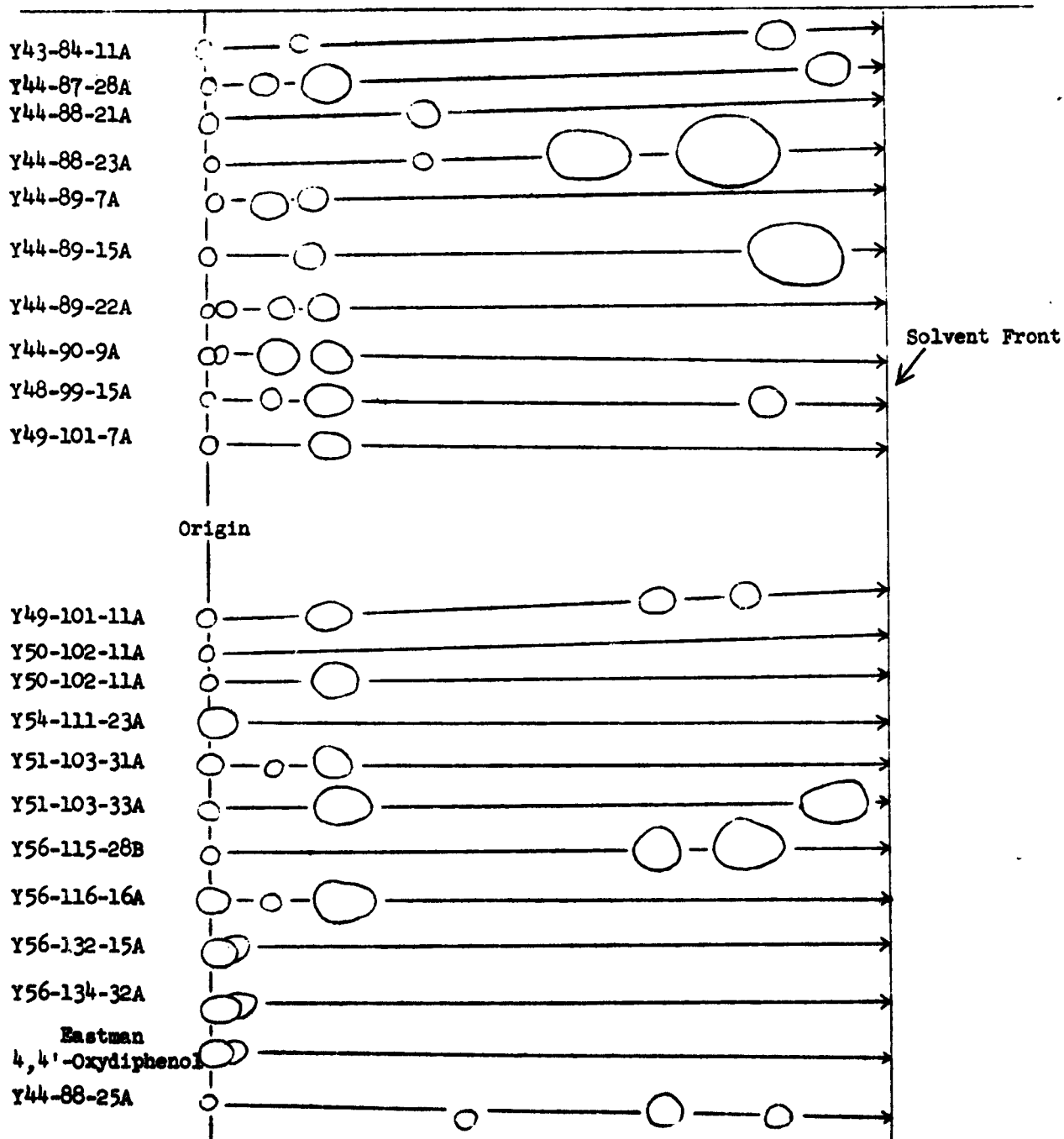
* Desired hydroxyl # is 554.9 (equivalent KOH)

NOTE: It is believed that a considerable portion of the desired product was lost in the alumina bed even though the bed was flushed three times with hot acetone.

FIGURE II

Thin Layer Chromatography

Representative Plate Developed with Benzene



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